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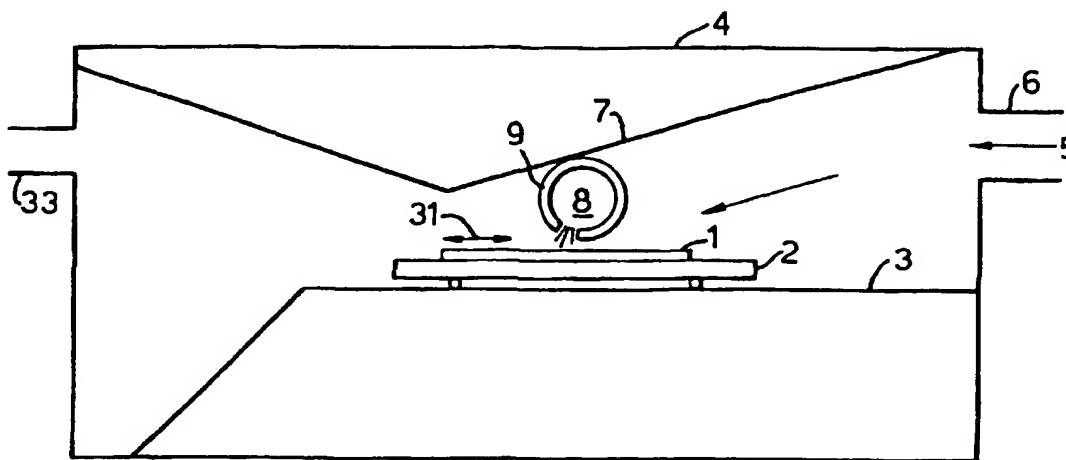
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(54) Title: METHOD AND APPARATUS FOR EPITAXIALLY GROWING A MATERIAL ON A SUBSTRATE



(57) Abstract

A method of epitaxially growing a material on a substrate (1). The method comprises separately heating precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately to the region and which combine at the region.

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METHOD AND APPARATUS FOR EPITAXIALLY GROWING
A MATERIAL ON A SUBSTRATE

The invention relates to a method and apparatus for
5 epitaxially growing a material on a substrate.

Conventional methods typically use a metalorganic
chemical vapour phase deposition (MOCVD) epitaxial reactor.
In such a reactor, the precursors containing the elements
which are to be grown are supplied to a substrate such as
10 a wafer supported on a wafer carrier which is heated. Heat
is conveyed to the precursors which crack or dissociate
into nascent atoms which then combine on the surface of the
substrate. In a typical example, trimethylgallium and
ammonia, together with hydrogen, are supplied to the
15 substrate to form a gallium nitride layer.

A problem with known reactors is that the precursors
have cracking temperatures which are much higher than the
ideal growth temperatures. For example, ammonia typically
requires temperatures up to 1000°C or more to crack
20 efficiently while the ideal growth temperature is in the
order of 650°C.

The problem with conventional reactors is that the
wafer carrier will maintain the substrate at a relatively
constant temperature which typically will be chosen to be
25 the ideal growth temperature, in this example 650°C. Group
V precursors such as ammonia are only very inefficiently
cracked at this temperature.

JP-58125698 and JP-58140391 disclose methods of
growing InP on a substrate using gaseous mixtures of
30 precursors and decomposition species. JP-04074858 and JP-
05335622 disclose alternative high vacuum deposition
processes in which the decomposition species are reacted in
a precursor atmosphere. Here the problem is addressed by
decomposing one precursor at a remote location and then
35 mixing the decomposition species with the other precursors
and their decomposition species prior to deposition.
However, problems still remain due to the occurrence of

undesirable reactions in the mixture which reduce the overall efficiency of the process.

In accordance with one aspect of the present invention a method of epitaxially growing a material on a substrate comprises separately heating precursors to their respective decomposition temperatures at or adjacent to a region of the substrate to generate species which are supplied separately to the region and which combine at the region.

In contrast to the known methods, we separately heat the precursors to their respective decomposition temperatures at or adjacent this region, the region constituting a growth region in which the species combine. In this way, each precursor can be heated to its most efficient decomposition (cracking) temperature, while carrying out this process adjacent to the region minimises the risk of nascent atoms recombining before reaching the substrate surface.

The species formed by the decomposition of the precursors are highly reactive and rapidly form more stable products. The probability of a species being involved in further reactions is a function of time and concentration. By decomposing the precursors in the vicinity of the growth region, the species formed are encouraged to combine at the growth region on the substrate, rather than forming undesirable reaction products. The decomposition of the precursors in close proximity to the growth region reduces the time period in which unfavourable reactions can occur.

Preferably the species from each precursor are supplied separately to the region in a sequential manner. The species may be supplied separately to the region by moving the substrate, to cause the movement of the region with respect to the locations at which decomposition of the precursors occurs. Typically, at least one of the precursors will be supplied separately to the region as a gas stream.

Preferably the species may be chosen from the Group III and Group V elements. Alternatively the Group IV elements may be used such as Silicon and Carbon.

5 In the preferred example, one of the precursors, preferably the one with the lowest decomposition temperature, is heated to its cracking temperature by heating the substrate. In this preferred example, another of the precursors is heated to its cracking temperature at a location adjacent the region. Thus, the substrate could
10 be heated in the range 550-800°C, for example 650°C, while the other precursor is heated to its optimum cracking temperature either directly or in the presence of a catalyst. Typically this temperature is in the range 400-1800°C.

15 In accordance with a second aspect of the present invention, apparatus for epitaxially growing a material on a substrate comprises a chamber containing a substrate support, the chamber having a first inlet for supplying a first precursor and a second inlet, separate from the first
20 inlet, for supplying a second precursor; and first and second heating means for separately heating the first and second precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which combine at the region.

25 Preferably, the second inlet is formed in a supply conduit located adjacent to the substrate support. This provides a convenient way of bringing the second precursor close to the substrate.

30 The second inlet can take a variety of forms including for example a circular hole or the like but is preferably in the form of an elongate slot.

The second heating means is conveniently provided in or adjacent the slot although it could be spaced upstream of the slot.

35 The supply conduit will typically be made of a refractory material such as quartz, SiN or alumina while

the second inlet preferably defines a jet aperture having a transverse dimension of for example 2mm.

Preferably, the second inlet defines an outlet direction for the precursor which is at an acute angle to the substrate support since this will create a venturi effect which will encourage other gases such as hydrogen supplied from a remote source to pass under the conduit.

The second heating means is typically in the form of a heating wire made for example of iron, nickel, aluminium, platinum or their alloys, and in particular Platinum-Rhodium. The wire will typically be coiled.

In most applications, it is desired to extend the epitaxial growth to a relatively large region of the substrate. This is most conveniently achieved by moving the region across the substrate, for example by providing means for causing relative movement between the substrate support and at least one of the inlets.

Furthermore, the invention is not limited to the provision of a single region upon a single substrate. For example, a plurality of supply conduits may be provided for supplying the same or different precursors to regions on the substrate, the conduits and substrate support being relatively movable to bring the conduits into alignment with different regions. The supply conduits may be arranged to supply precursors separately and sequentially to the region.

Some examples of methods and apparatus according to the invention will now be described with reference to the accompanying drawings, in which:-

Figure 1 is a schematic cross-section through the apparatus of a first example;

Figure 2 is an enlarged, cross-section through part of the apparatus shown in Figure 1;

Figure 3 is an underneath plan of part of the apparatus shown in Figure 1;

Figure 4 is a schematic, cross-section through the apparatus of a second example;

Figure 5 is a schematic plan of a second example; and Figure 6 is a schematic plan of a third example.

The reactor shown in Figure 1 comprises a reaction chamber 4 within which is provided a substrate carrier (susceptor) 3 which supports a substrate support (wafer carrier) 2. In Figure 1, a substrate (wafer) 1 is shown in place on the substrate support 2. The substrate carrier 3 may be a graphite block which is heated in a conventional manner by for example induction, infra-red or resistance heating methods.

A first inlet 6 is provided in a side wall of the reaction chamber 4 while a gas deflector 7 is mounted within the reaction chamber to direct precursor gases 5, entering through the inlet 6, towards the wafer 1.

An injector conduit 9 extends transversely across the reaction chamber 4 and is in the form of a refractory tube of for example quartz having an elongate slot 10 forming a jetting aperture extending along its length above the wafer carrier 2 (see Figure 2).

A straight wire or coil 11 is supported within the slot 10 to form a heating element.

In use, a substrate 1 is placed on the substrate support 2, the reaction chamber 4 is closed and then the pressure within the chamber adjusted as appropriate, typically in the range 5-1000 torr. Typically, the wafer material is chosen to promote epitaxial growth of the material and can include sapphire, GaN, GaAs, SiC or ZnO.

One or more reactive precursor gases 5 are introduced into the chamber through the inlet 6. Examples of the reactive gases include trimethylgallium (for Ga deposition), trimethylindium (for indium deposition) and so on depending upon the required composition of the deposited layer. Such a mixture may also contain precursors for semiconductor dopants. A gas such as hydrogen is also added to the mixture 5 to stabilise the radicals produced on dissociation of the precursor.

The substrate carrier 3 is heated such that the substrate 1 and the substrate support 2 are also heated due to their proximity to the substrate carrier 3 attaining a suitable growth temperature for the substrate 1 i.e. a temperature at which the precursor 5 introduced through the inlet 6 will most efficiently crack. A suitable temperature for the growth of GaN is in the range 600-800°C.

A second precursor gas 8, in this example ammonia, is supplied along the conduit 9 under pressure so that it exits through the jetting slot 10 past the heating wire 11. The wire 11 is heated to a temperature typically in the range 400-1800°C suitable for achieving optimum cracking of ammonia to generate N and H radicals. It will be noted in Figure 2 that the slot 10 is angled relative to a normal to the wafer 1 so as to cause gases introduced through the inlet 5 to be drawn under the conduit 9 as shown by an arrow 30 as a result of a venturi effect.

The precursor gas 5, upon reaching the substrate 1, will be cracked in a conventional manner and the resultant species, for example Ga atoms, will combine with nitrogen radicals from the gas 8 to initiate epitaxial growth of gallium nitride on the substrate 1.

The wire 11 is preferably made from a catalytic material such as a platinum-rhodium alloy and may be heated in the case of this alloy, to a temperature typically in the range 600-700°C. This can be achieved by electrical resistance heating if the element takes the form of a filament wire. The distance between the wire 11 and the substrate 1 is small to allow the maximum quantity of cracked product from the precursor 8 to react with the precursor mixture 5 at the substrate surface. This optimum distance is also influenced by the flow rate of the mixture 5, typical conditions for ammonia in the production of GaN being a distance of around 5mm for a flow velocity of 0.35ms⁻¹.

The jetting of the second precursor gas species on to the region of the substrate provides an advantage in that the jet acts as a gas shield which sweeps away any of the precursor gas 5 and its decomposition products that are not attached to the substrate. This allows the nitrogen and hydrogen radicals to reach the substrate surface in greater concentrations as they are not mixed with the precursor gas 5 or its decomposition species before reaching it. This example therefore allows the species from each precursor to separately contact the surface of the substrate.

In order to control the area of growth, the substrate support 2 is movably mounted on the substrate carrier 3 and can be moved (by means not shown) in the directions indicated by an arrow 31 beneath the conduit 9. This movement allows the species from each precursor to separately reach the substrate surface sequentially, in any order, without mixing with the other precursor or its decomposition species.

This process can then be repeated to enable further layers to be laid down on the wafer so producing multi-layered wafers containing materials of dissimilar dimensions, dopants or compositions.

Waste gases finally exit through an outlet 33.

An application for this invention is the production of microwave and optoelectronic devices. These devices are built up layer upon layer from different materials with different electrical properties. The configuration of these layers determines whether the device is for microwave or light emission i.e. a Field Effect Transistor or a Light Emitting Diode. To achieve optimum performance the layers have to be grown where possible at the most ideal temperature. This temperature will vary depending upon the material used and the device being fabricated. The advantage of this invention is that the growth temperature of the material is not compromised by the necessity to have a high temperature to crack the precursor materials such as ammonia. Advantageously the invention also provides the

possibility of using precursors that would otherwise be incompatible in the gas phase such as dicyclopentadienylmagnesium, as these would otherwise form low vapour pressure adducts with group V species, for example from ammonia.

A second example of the present invention is shown in Figure 4. In this case the reaction chamber 4' comprises a tubular quartz outer cell 60 of substantially circular cross-section attached to a cell block end 61. Within the outer cell 60 is positioned an inner cell 65 of substantially square tubular section. A removable molybdenum carrier plate 66 is seated within inner cell 65. The carrier plate 66 extends along the base of the inner cell 65. The substrate carrier 3 equipped with heating means (not shown) rests upon the carrier plate 66. Additionally a self-supporting quartz gas deflector 7' is also positioned upon the carrier plate 66. An exit vent 67 for the gases is provided through the cell block end 61.

The injector conduit 9 enters the cell through the cell block end 61 and in contrast to the example shown in Figure 1, lies in a direction approximately parallel to that of the major gas flow (indicated by an arrow 68 in Figure 4) and extends only halfway across the substrate 1. The elongate slot 10 and heating element 11 are of appropriate dimensions to produce deposition in a region between the outer edge and centre of the substrate. In this case the substrate support 2 is positioned within a recess 70 provided in the substrate carrier 3. The substrate support itself also contains a recess 71 into which the substrate 1 is placed during deposition. The dimensions of both recesses and the substrate are such that the upper surfaces of the substrate 1 and substrate support 2 are substantially flush with that of the substrate carrier 3. The substrate support is mounted upon a shaft 75 which enters the cell from beneath and passes through holes in the substrate carrier 3, the carrier plate 66 and the cell walls 60, 65. The cell walls 60, 65 are equipped

with suitable gas seals to maintain the cell leak tight. The lower end of the shaft is attached to a motor 80 or other suitable rotation means.

During operation both the outer and inner cells are
5 evacuated in order to reduce the pressure differential across the inner cell walls 65. As in the previous example, one or more precursor gases enter the inner cell from an inlet on the right of the figure (not shown). Deposition of the material also occurs in a similar manner.
10 However, here the rotation of shaft 75 by the motor (shown by arrow 76 in Figure 4) causes relative rotational rather than reciprocal motion with respect to the injector conduit.

In this example the injector conduit 9 and shaft 75
15 are detachable from the apparatus, allowing the slidable removal of the carrier plate 66, gas deflector 7' and substrate carrier assembly.

Figure 5 shows apparatus for growth of materials upon a plurality of substrates 17 mounted upon a heated
20 susceptor 18 in a chamber (not shown). One or both of the conduits 19 and the susceptor 18 are rotatable. Layers of a semiconducting wafer may be deposited sequentially using a plurality of precursor supply conduits 19, similar to the conduit 9, radiating from the centre of the susceptor. In
25 one example, the supply conduits are grouped as shown so as to bring precursors for both reacting species to a single region simultaneously. Substrates 17 are successively brought into close proximity to supply conduits 19 by relative movement of the conduits 19 and substrates 17. In
30 this example, hydrogen, nitrogen, argon, or another suitable sweep gas is introduced to the system at the centre of the substrate support, flowing radially outwards rather than being supplied directly with one of the precursors. This flow sweeps away the spent reaction
35 products.

In alternative example shown in Figure 6, conduits 19a and 19b individually supply precursors sequentially to the

growth region, a rapid relative movement between each substrate and each precursor conduit ensuring that only a short time elapses between the delivery of each species. The efficiency of the deposition process is therefore
5 increased as in each case the species from the precursors are allowed to reach the substrate without reacting with species from other precursors or the precursors themselves.

The conduits 19a and 19b supply the precursors from around the periphery of an annular susceptor. The
10 substrates are positioned circumferentially around the surface of the annular susceptor. It will be appreciated that one or more of the conduits could be equipped with means to decompose one or more of the precursors supplied.

In this example each of the conduits is equipped with
15 heated wires to decompose the separate precursors at their respective temperatures, conduits 19a supplying the Group V precursor ammonia and conduits 19b supplying the Group III precursor trimethylgallium. The substrates 17 are also heated by similar methods to those in the previous
20 examples, the heating ensuring the uniformity of the deposited materials.

This arrangement is particularly advantageous in that the apparatus can be scaled up to manufacture devices in large quantities without altering the gas flow on chemistry
25 in the vicinity of each substrate. This may be achieved by increasing the radius of the annular susceptor 18 allowing more substrates to be positioned around its circumference, along with providing more conduits 19a and 19b as appropriate. In addition, substrates of different sizes
30 may also be used.

Although not shown in Figure 6, a radial flow of hydrogen is also provided in this example. Alternatively other gases such as nitrogen or argon could be used.

CLAIMS

1. A method of epitaxially growing a material on a substrate, the method comprising separately heating
5 precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately to the region and which combine at the region.
2. A method according to claim 1, wherein the species
10 from each precursor are supplied separately to the region in a sequential manner.
3. A method according to claim 1 or claim 2, wherein the species are supplied separately to the region by the relative movement of the substrate to cause the movement of
15 the region with respect to the locations at which decomposition of the precursors occurs.
4. A method according to any of the preceding claims, wherein at least one precursor is supplied separately to the region as a gas stream.
- 20 5. A method according to any of the preceding claims, wherein the species are chosen from the Group III and Group V elements.
6. A method according to any of claims 1 to 4, wherein the species are chosen from the Group IV elements.
- 25 7. A method according to claim 5, wherein the species comprise Gallium and Nitrogen.
8. A method according to claim 6, wherein the species comprise Carbon and Silicon.
9. A method according to claim 7, wherein one of the
30 precursors is ammonia.
10. A method according to any of the preceding claims, wherein the substrate comprises a semiconductor such as Gallium-Arsenide.
11. A method according to any of the preceding claims,
35 wherein one of the precursors is heated to its decomposition temperature by heating the substrate.

12. A method according to claim 6, wherein the substrate is heated to the decomposition temperature of the precursor with the lower decomposition temperature.
13. A method according to claim 11 or claim 12, wherein
5 the substrate is heated to a temperature in the range 550-800°C.
14. A method according to any of the preceding claims, wherein one of the precursors is heated to its decomposition temperature at a location adjacent the
10 region.
15. A method according to claim 14, wherein the precursor is heated to a temperature in the range 400-1800°C.
16. A method according to any of the preceding claims, further comprising moving the region across the substrate.
- 15 17. Apparatus for epitaxially growing a material on a substrate, the apparatus comprising a chamber containing a substrate support, the chamber having a first inlet for supplying a first precursor and a second inlet, separate from the first inlet, for supplying a second precursor; and
20 first and second heating means for separately heating the first and second precursors to their respective decomposition temperatures at or adjacent a region of the substrate to generate species which are supplied separately to the region and which combine at the region.
- 25 18. Apparatus according to claim 17, wherein the second inlet is formed in a supply conduit located adjacent the substrate support.
19. Apparatus according to claim 18, wherein the second inlet is in the form of an elongate slot.
- 30 20. Apparatus according to claim 18 or claim 19, wherein the second heating means is provided in or adjacent the slot.
21. A method according to any of claims 17 to 20, wherein the second heating means is in the form of a heating wire.
- 35 22. Apparatus according to any of claims 17 to 21, wherein the first heating means is located at a position to heat the substrate support.

23. Apparatus according to any of claims 17 to 22, further comprising means for causing relative movement between the substrate support and at least one of the inlets.

5 24. Apparatus according to claim 23, when dependent on at least claim 18, wherein a plurality of supply conduits are provided for supplying the same or different precursors to regions on the substrate, the conduits and substrate support being relatively movable to bring the conduits into alignment with different regions.

10 25. Apparatus according to claim 24, wherein the supply conduits are arranged to supply precursors separately and sequentially to the region.

15 26. Apparatus according to claim 23 wherein the relative movement between the substrate support and at least one of the inlets is in a transverse manner.

27. Apparatus according to claim 23 wherein the relative movement between the substrate support and at least one of the inlets is in a rotational manner.

Fig.1.

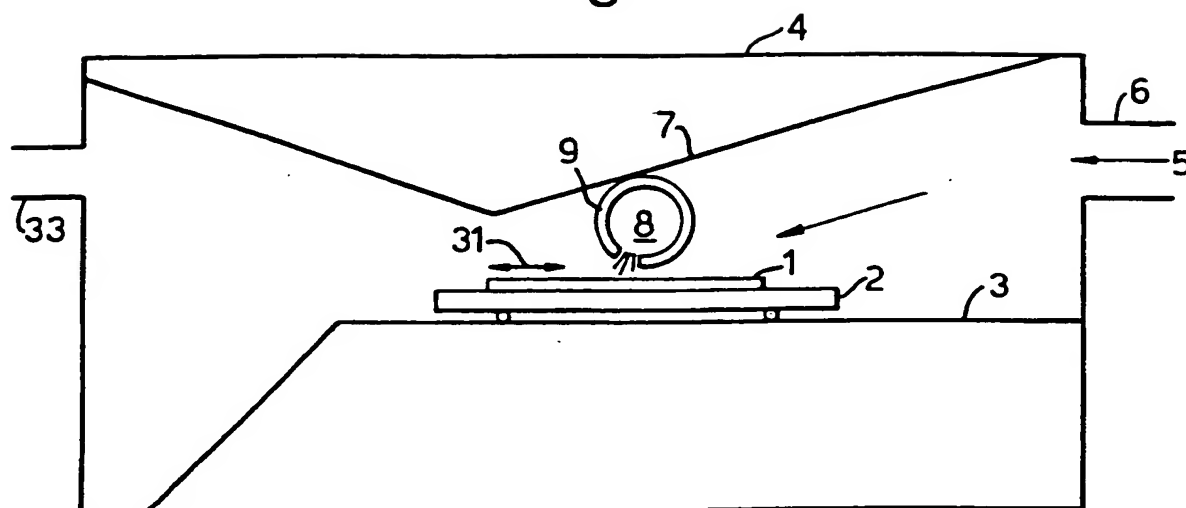


Fig.2.

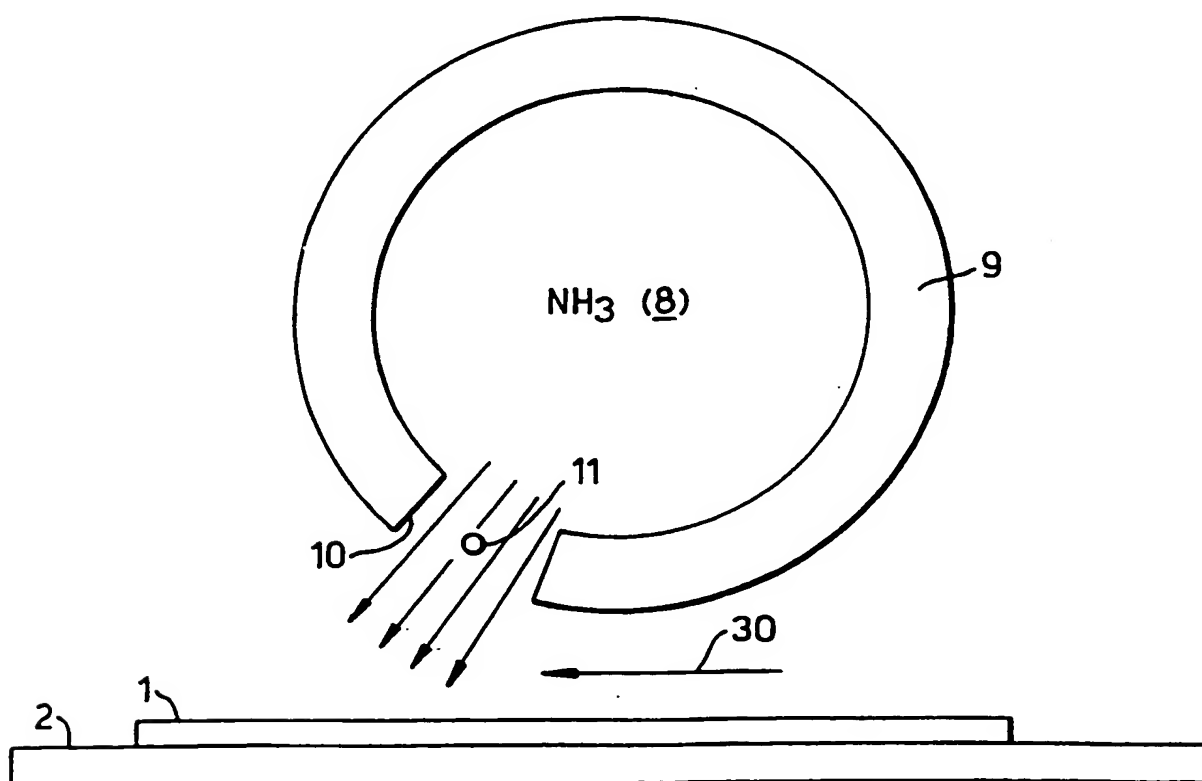


Fig.3.

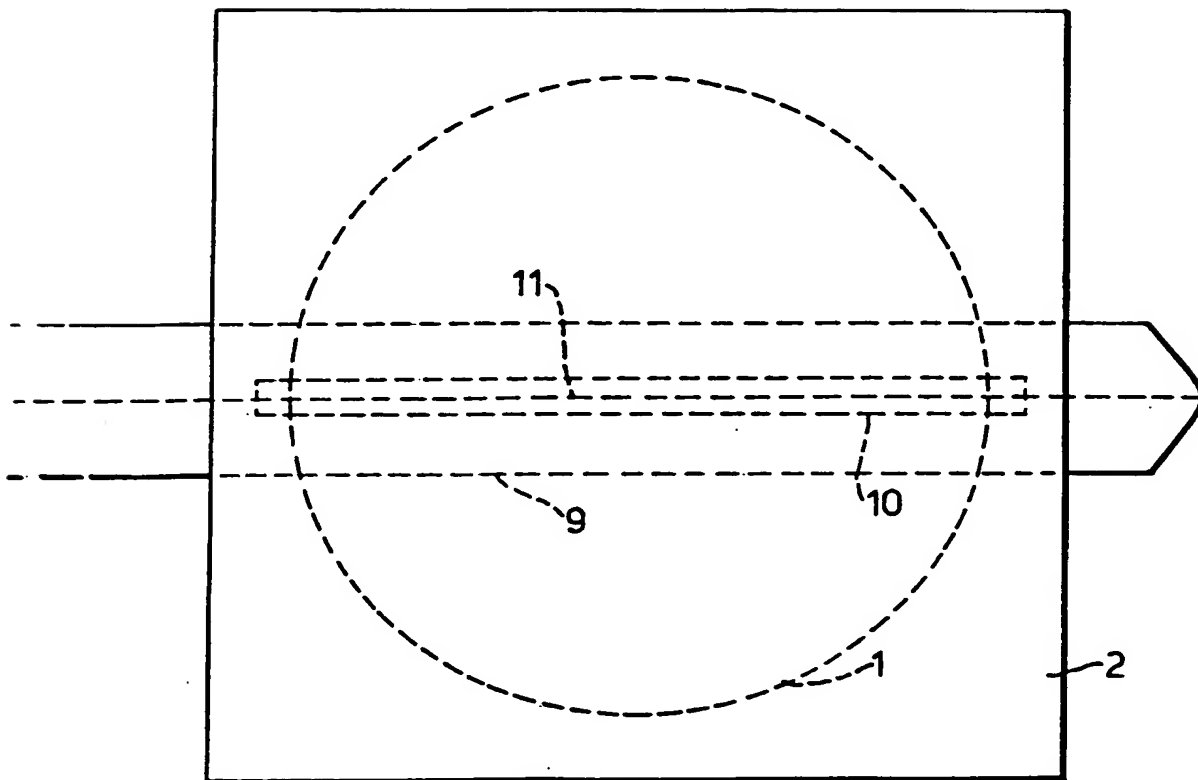


Fig.5.

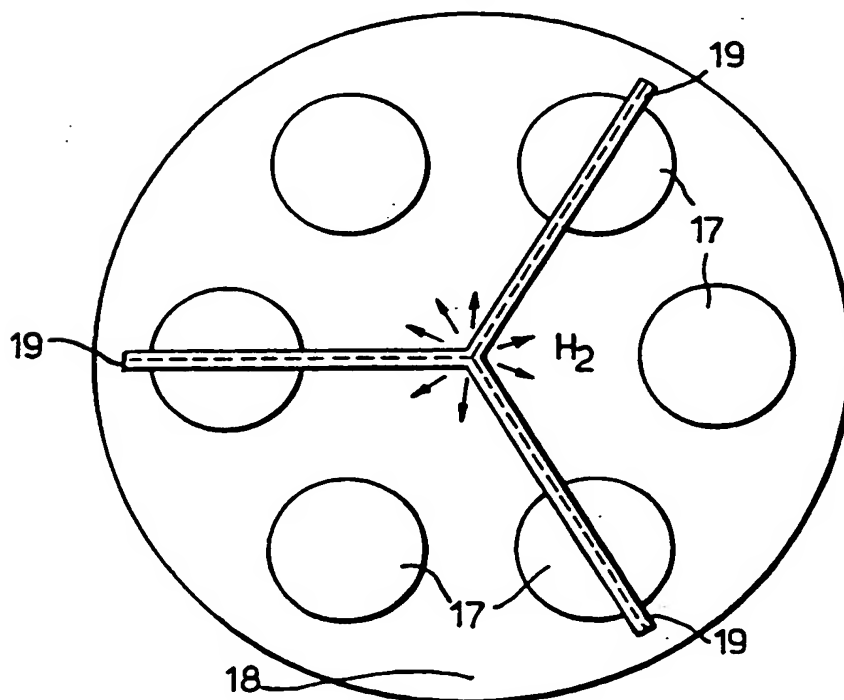


Fig.4.

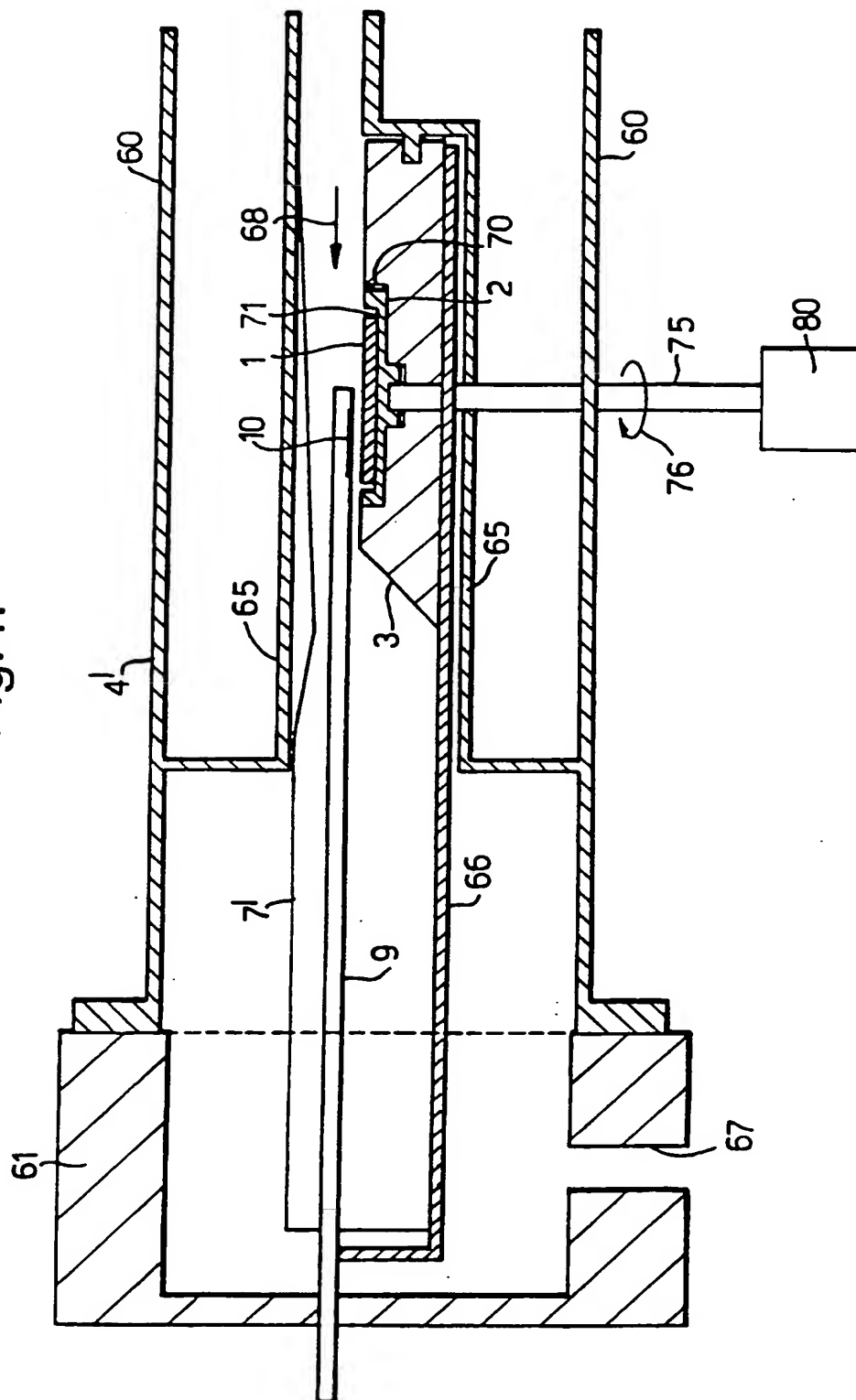
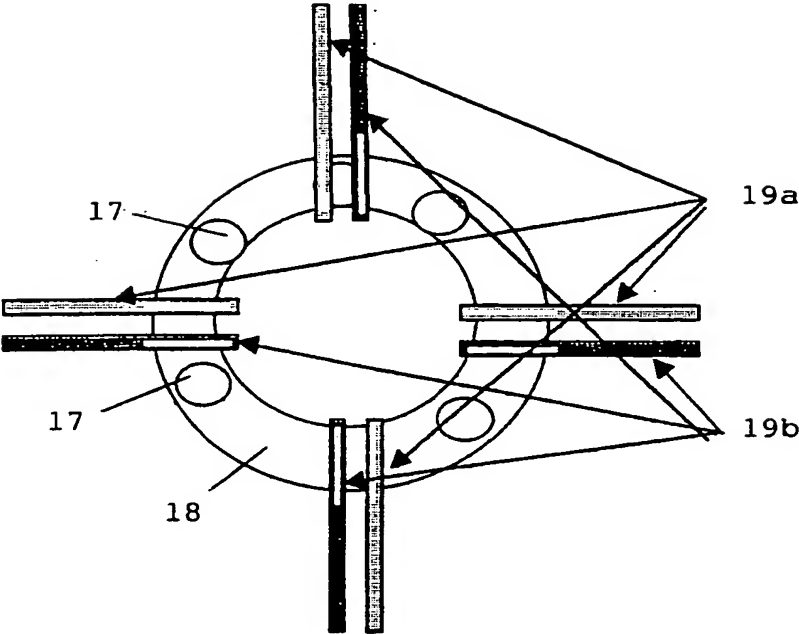


FIGURE 6



INTERNATIONAL SEARCH REPORT

Inventor's Name Application No

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A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C30B25/14 C30B25/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C30B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

International Application No

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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